Designation: C 1608 – 07

# Standard Test Method for Chemical Shrinkage of Hydraulic Cement Paste[1]

This standard is issued under the fixed designation C 1608; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

## 1. Scope*

1.1 This test method measures the internal (absolute) volume change of hydraulic cement paste that results from the hydration of the cementitious materials. This volume change is known as *chemical shrinkage*.

1.1.1 Procedure A, volumetric method.

1.1.2 Procedure B, the density method.

1.2 The values stated in SI units are to be regarded as the standard.

1.3 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* (**Warning**—Fresh hydraulic cementitious mixtures are caustic and may cause chemical burns to skin and tissue upon prolonged exposure.[2])

## 2. Referenced Documents

2.1 *ASTM Standards:* [3]

C 186 Test Method for Heat of Hydration of Hydraulic Cement

C 188 Test Method for Density of Hydraulic Cement

C 219 Terminology Relating to Hydraulic Cement

C 305 Practice for Mechanical Mixing of Hydraulic Cement Pastes and Mortars of Plastic Consistency

C 511 Specification for Mixing Rooms, Moist Cabinets, Moist Rooms, and Water Storage Tanks Used in the Testing of Hydraulic Cements and Concretes

C 670 Practice for Preparing Precision and Bias Statements for Test Methods for Construction Materials

C 1005 Specification for Reference Masses and Devices for Determining Mass and Volume for Use in the Physical Testing of Hydraulic Cements

## 3. Terminology

3.1 *Definitions:*

3.1.1 *chemical shrinkage*, *n*—the absolute (internal) volume change accompanying the hydration of cement, due to the fact that the cement hydration products occupy less physical volume than the reactants.

3.1.2 All other terms are as defined in Terminology C 219.

## 4. Significance and Use

Numerous properties of cementitious materials are controlled by their initial hydration rate. Examples include early-age strength development, heat release, and crack resistance. One direct and convenient measure of this initial hydration rate is provided by the measurement of the chemical shrinkage of the cement paste during its hydration. As cement hydrates, the hydration products occupy less volume than the initial reacting materials (cement and water). Due to this volume change, a hydrating cement paste will sorb water from its immediate surroundings, when available. At early times, this sorption is in direct proportion to the amount of hydration that has occurred.[4] This method is based on the one developed by Geiker.[5] The results are relevant to understanding the hydration behavior of cements. This method does not measure the bulk volume changes (autogenous shrinkage) associated with chemical shrinkage nor the cracking potential of concretes produced with the evaluated cement.

## 5. Apparatus

5.1 *Devices for Determining Mass*, conforming to the requirements of Specification C 1005 and evaluated for precision and accuracy at a total load of 100 g.

5.2 *Constant Temperature Water Bath*—a water bath capable of maintaining a temperature of 23.0 ± 0.5 °C, with a

[1] This test method is under the jurisdiction of ASTM Committee C01 on Cement and is the direct responsibility of Subcommittee C01.31 on Volume Change.

Current edition approved June 15, 2007. Published July 2007. Originally approved in 2005. Last previous edition approved in 2006 as C 1608 – 06.

[2] See the section on Safety, Manual of Cement Testing, *Annual Book of ASTM Standards*, Vol. 04.01.

[3] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[4] L.J. Parrott, M. Geiker, W.A. Gutteridge, and D. Killoh, "Monitoring Portland Cement Hydration: Comparison of Methods," Cement and Concrete Research, Vol. 20, 919-926, 1990.

[5] M. Geiker, "Studies of Portland Cement Hydration: Measurements of Chemical Shrinkage and a Systematic Evaluation of Hydration Curves by Means of the Dispersion Model," Ph.D. Thesis, Technical University of Denmark, Copenhagen, Denmark, 1983.

***A Summary of Changes section appears at the end of this standard.**

sufficient capacity to hold the specimens being evaluated. To avoid evaporative cooling, the surface of the water in the bath shall be covered with floating plastic balls or fitted with an insulated lid.

5.3 *Timing Device*—Clock that can measure time to the nearest minute.

5.4 For procedure A

5.4.1 *Capillary Tube*—A graduated glass capillary tube with graduations of 0.01 mL or smaller, and typically a capacity of 1.0 mL.

5.4.2 *Small Glass Vials (e.g., 22-mm diameter and 55-mm height) with Rubber Stoppers* that fit tightly into the glass vials and have a hole placed in each stopper with the graduated capillary tube inserted through the hole (as shown in Fig. 1). Fix the capillary tube in the stopper using a two-component epoxy or other suitable adhesive applied at the stopper's top and bottom surfaces.

5.5 For Procedure B

5.5.1 *Density bottle*, glass, capacity approximately 20 ml with internally conical glass stopper as shown in Fig. 2.

## 6. Reagents and Materials

6.1 Paraffin oil.

6.2 De-aerated water (prepared by boiling water and sealing it in a closed container before it has cooled.)

## 7. Procedure

7.1 *Preparation of Cement Paste*—Prepare the cement paste in accordance with the proportions and procedure described in Test Method C 186 (Note 1). The pastes shall be prepared in a mixing room meeting the temperature and humidity requirements outlined in Specification C 511. Record to the nearest minute the time when the water first contacts the dry cement powder.

NOTE 1—Other mixing procedures such as mixing in a Hobart mixer (see Practice C 305) or kneading by hand in a sealed plastic bag may be used. The standard paste mixture uses 150 g of cement and 60 ml of water (water-cement ratio of 0.40). Adjust the volume of paste mixed to match the mixing equipment used. Other water-cement ratios may be used as long as they are stated in the test report; higher water-cement ratios may produce significant bleeding of the cement paste which will influence the results (by changing the effective water-cement ratio, etc.); lower water-cement ratios may lead to difficulties in preparing a fully compacted, homogeneous paste for subsequent evaluation and self-desiccation may occur.

7.2 Prepare a minimum of two replicate specimens as described below for either Procedure A or Procedure B.

7.3 Procedure A

7.3.1 Determine the mass of each empty glass vial to the nearest 0.01 g.

7.3.2 Carefully place the prepared cement paste into the glass vial to achieve a paste height between 5 mm and 10 mm in the vial. Consolidate the paste in the vial by tapping the vial on a laboratory countertop, or placing it on a vibrating table, or some similar procedure.

7.3.3 Determine the mass of each glass vial with the cement paste to the nearest 0.01 g.

7.3.4 Carefully, without disturbing the cement paste, add clean, de-aerated water to fill the glass vial to the top.

7.3.5 Place the rubber stopper with the inserted capillary tube tightly into the glass vial. Be careful to avoid the entrapment of air bubbles when the bottom rubber stopper surface encounters the water in the glass vial. As the rubber stopper is inserted, the water level in the graduated capillary tube will rise. Optimally, the water level should rise near to but not beyond the top mark of the graduations on the capillary tube. If the water level is not near enough to the top mark, clean, de-aerated water can be added via the top of the capillary

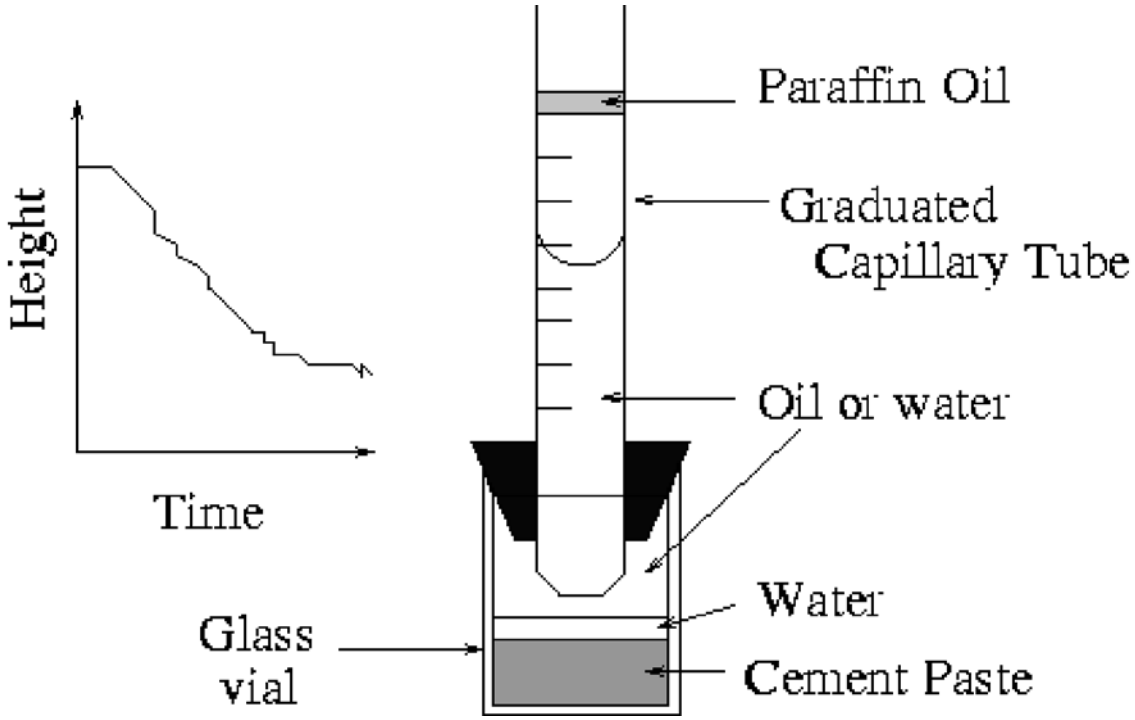


**FIG. 1 Illustration of One Experimental Setup for Monitoring Chemical Shrinkage of Hydrating Cement Paste Using Procedure A.**

tube to achieve the desired initial water height. Once the rubber stopper has been fitted to the vial, the completed assembly shall be handled only by the vial (not by the capillary tube) to avoid loosening of the rubber stopper which will result in an apparent volume change of the specimen.

7.3.6 Place a drop of paraffin oil in the top of the graduated capillary tube to minimize water evaporation from the tube during the testing period.

7.3.7 Place the prepared specimens in the constant temperature water bath at 23 °C such that the tops of the glass vials are just above the water level in the bath. Maintain the temperature of the laboratory at 23 ± 2 °C. Record the time and initial level (height) of water, to the nearest 0.0025 mL, in the capillary tubes.

7.3.8 Periodically (every 30 min or every hour, as convenient) record the time to the nearest minute and water level in the capillary tubes to the nearest 0.0025 mL for a total period of at least 24 h. After the first 8 h, the recording intervals can be lengthened to 8 h or more to avoid taking readings during the night, as long as a 24 h reading is obtained the following day. Be sure to take a reading 1 h after the paste was first mixed to use as a zero point in all calculations (this allows time for the specimen to achieve temperature equilibrium within the water bath).

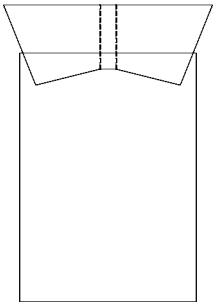

**FIG. 2 Illustration of a Density Bottle for Use in Procedure B.**

7.4 Procedure B

7.4.1 Determine the mass of each empty density bottle to the nearest 0.0001 g.

7.4.2 Carefully place the prepared cement paste into the density bottle to achieve a paste height between 5 mm and 10 mm in the bottle. Consolidate the paste in the bottle by tapping the bottle on a laboratory countertop, or placing it on a vibrating table or some similar procedure. Determine the mass of the density bottle with the consolidated cement paste to the nearest 0.0001 g.

7.4.3 Carefully, without disturbing the cement paste, add clean, de-aerated water to fill the bottle to the top.

7.4.4 Place the stopper tightly into the bottle. Be careful to remove any entrapped air bubbles when the bottom of the stopper encounters the water in the bottle. Add clean, de-aerated water via the perforated stopper to fill the bottle and stopper to excess (leave a bead of water on the top of the stopper to overfill the capillary tube). The filled assembly shall be handled only by the bottle (not by the stopper) to avoid loosening the stopper, which will result in an apparent change in the specimen, and not with bare fingers (e.g., wear latex gloves) which will cause an increase in mass due to transfer of oil.

7.4.5 Remove the excess water from the top of the stopper using absorbent paper, wiping quickly to avoid sucking water out of the capillary tube.

7.4.6 Immediately determine the mass of the density bottle filled with water to the top of the capillary tube. Determine the mass to the nearest 0.0001 g.

7.4.7 Place the filled density bottles in the constant temperature water bath at 23 °C such that the tops of the stoppers are just above the water level in the bath.

7.4.8 At 1 h after the paste was first mixed, remove the density bottle from the water bath and wipe dry, fill to excess with water, remove the excess and determine the mass to the nearest 0.0001 g.

7.4.9 Periodically (every 30 or 60 min, as convenient) for a period of at least 24 h, remove the density bottle from the water bath and wipe dry, fill to excess with water, remove the excess and determine the mass to the nearest 0.0001 g. After the first eight hours, the recording intervals can be lengthened to 8 h or more to avoid taking readings during the night, as long as a 24 h reading is obtained the following day.

## 8. Calculation

8.1 The chemical shrinkage is computed as the measured mL of sorbed water per gram of cement in the paste specimen. The mass of cement powder in the vial is given by:

$$M_{cement} = \frac{(M_{vial+paste} - M_{vialempty})}{\left(1.0 + \frac{w}{c}\right)} \quad (1)$$

where

$M_{cement}$ = mass of cement in the vial (g),
$M_{vial+paste}$ = mass of the glass vial with the added cement paste (g),
$M_{vialempty}$ = mass of the empty vial (g),
$w/c$ = water-cement ratio by mass of the prepared paste (e.g., 0.40) and a density of 1000 kg/m$^3$ is assumed for water.

8.2 Procedure A

8.2.1 The chemical shrinkage per unit mass of cement at time t is computed as:

$$CS(t) = \frac{[h(t) - h(60\text{min})]}{M_{cement}} \quad (2)$$

where

$CS(t)$ = chemical shrinkage at time t (mL/g cement) (Note 2).
$h(t)$ = water level in capillary tube at time t (mL)

NOTE 2—If it is desired to report the chemical shrinkage per unit volume of cement powder, the values from Eq 2 can be converted to mL/mL cement using the density of the cement powder determined using Test Method C 188.

8.3 Procedure B

8.3.1 The chemical shrinkage per unit mass of cement at time t is computed as:

$$CS(t) = \left(\frac{[M(t) - M(60\text{min})]}{M_{cement}}\right)/\rho W \quad (3)$$

where:
$CS(t)$ = chemical shrinkage at time t (mL/g cement)
$M(t)$ = mass of filled density bottle at time t (g)
$\rho W$ = density of water (mL/g) (0.99754 at 23 °C)

## 9. Report

9.1 Report the following:

9.1.1 Date and time paste specimen is prepared;

9.1.2 Cementitious material sources and names;

9.1.3 Mixing and compaction procedure employed and water-cement ratio by mass;

9.1.4 Mass of each empty vial and mass of each vial with cement paste;

9.1.5 A table of the water level in each capillary tube or the mass of each filled density bottle vs. time;

9.1.6 A table and/or plot of the chemical shrinkage per gram of cement (reported to the nearest 0.0001 mL/g) vs. time.

## 10. Precision and Bias

10.1 *Precision*—The precision statements for this test method are listed in Table 1 and are based on the results obtained in an interlaboratory study described in Research Report C01-1010.[6]

10.2 *Bias*—Since there is no accepted reference material suitable for determining the bias of this procedure, no statement on bias is made.

## 11. Keywords

11.1 chemical shrinkage; hydration.

[6] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR: C01-1010.

**TABLE 1 Precision**

| | Standard Deviation 1s[A] | Acceptable Range of Test Results d2s[A] |
|---|---|---|
| Measurements made between 4 and 8 h after mixing | | |
| Repeatability | 0.00084 | 0.00236 |
| Reproducibility | 0.00090 | 0.00253 |
| Measurements made 24 h after mixing | | |
| Repeatability | 0.00193 | 0.00541 |
| Reproducibility | 0.00240 | 0.00672 |

[A] These numbers represent, respectively, the (1s) and (d2s) limits as described in Practice C 670.

## SUMMARY OF CHANGES

Committee C01 has identified the location of selected changes to this test method since the last issue, C 1608 – 06, that may impact the use of this test method. (Approved June 15, 2007)

(*1*) Revised 10.1 and added new Table 1.

Committee C01 has identified the location of selected changes to this test method since the last issue, C 1608 – 05, that may impact the use of this test method. (Approved November 1, 2006)

(*1*) Revised 7.3.4 and 7.4.3.